

# Characterization of Diamond-Like Film after Focused Ion Beam Processing

Yongqi Fu, Ngoi Kok Ann Bryan

**Abstract**—Optical properties (transmission and refractive index) and chemical structure (XPS) of diamond-like coating (DLC) film before and after focused ion beam milling (FIBM) process, were investigated in this paper. It is shown by our FIBM experiment that influence of FIBM on Optical properties and chemical structure of the DLC film is not apparent, especially in near-infra region. The film still can be used for optical applications. As an application example, diffractive optical element (DOE) was directly fabricated on the film by use of FIBM. Measured diffraction efficiency of the DOE is 73%, which is acceptable for conventional use.

**Index terms**—DLC, FIBM, optical properties, XPS

## I. INTRODUCTION

CHEMICAL vapor deposition (CVD) Diamond film with advantages of wide optical transmission windows, resistant to chemical attack and abrasion, and less resilient to hostile environments, bio-compatibility, makes it appealing and promising for optical system usage, e.g. protective coating in infra system [1–5]. It is especially superior to conventional optical materials in the application of integrated optical systems for delivery and broadband detection in micro-fluidic electrochromatography in which the integrated micro-optical elements can be protected by the diamond film due to its features of chemical inertness and biocompatibility [6,7]. Considering this, we can directly fabricate the micro-optical element on the diamond film in the integration system. However, it is difficult to use it fabricating conventional optical element due to limitation of diamond coating process which is difficult to realize large thickness ( $>2\text{ }\mu\text{m}$ ) coating because of large internal stress, which causes film crack or peel off. On the other hand, investigation of physical properties of the film, especially for optical properties after micromachining, is much concerned for designer and user of optical system. Few paper report variation of optical properties after processing till now, e.g. ion beam sputtering or  $\text{Ca}^+$  milling.

In this paper, we introduce a technique — focused ion beam milling (FIBM) for fabrication of micro-optical elements on diamond film. As an application example, we fabricate micro-diffractive optical element (DOE) with diameter of  $65\text{ }\mu\text{m}$  on the film. It is a direct fabrication method with features of one-step, isotropic etching, and non-selectivity of material. Because relief depth

of DOE is around  $1\text{ }\mu\text{m}$  for most glass material that is valid and practical thickness for diamond coating process. Therefore, the diamond film is suitable to be used for micromachining of DOE in the view of thickness limitation. Optical properties of the diamond film before and after FIBM is discussed in detail.

## II. SAMPLE PREPARATION

Diamond deposition was accomplished by standard hot filament CVD (HFCVD). The process parameters were listed as follows: 1%  $\text{CH}_4$  in  $\text{H}_2$ ; pressure,  $1\times 10^{-7}$  Torr; substrate temperature,  $2400\text{ }^\circ\text{C}$ ; filament substrate distance, 5 mm. Throughout the work a  $\text{CH}_4/\text{H}_2$  ratio of 100/1 was used at flow rates of 200 and 2 sccm, respectively, with a reactor pressure of  $\sim 40$  Torr. Thickness of the diamond film is  $1.5\text{ }\mu\text{m}$  with growth time of 25 h. Measured surface roughness (rms) of the diamond film is 31 nm (measured via atomic force microscopy (AFM) in the area of  $100\times 100\text{ }\mu\text{m}^2$ ).

The milling experiments were carried out by use of our FIB machine (Micrion 9500EX) with ion source of liquid gallium, integrated with scanning electron microscope (SEM), energy dispersion X-ray spectrometer (EDX) facilities and gas assistant etching (GAE) functions. This machine uses a focused  $\text{Ga}^+$  ion beam with energy of 50 keV, a probe current of 4 pA–19.7 nA and beam limiting aperture size of  $25\text{ }\mu\text{m}$ – $350\text{ }\mu\text{m}$ . For the smallest beam currents, the beam can be focused down to 7nm in diameter at full width and half maximum (FWHM). The milling process is performed under programming control, by means of varying the ion dose for different relief depth. UV-VIS transmittance spectrum (UV-VIS spectrophotometer) will be used to measure transmittance of the film before and after FIB processing. It requires illumination area larger than  $10\times 10\text{ mm}^2$ . The area is quite large for FIB milling (very time consuming for FIB machine). The maximum milling area of our machine is  $1\times 1\text{ mm}^2$ . We have to spell the area of  $10\times 10\text{ mm}^2$  by milling a  $10\times 10$  array. The spelling causes degradation of surface roughness uniformity, which will affect transmittance to a certain extent.

## III. RESULTS AND DISCUSSIONS

### A. XPS results comparison before and after FIB processing

Optical properties of the diamond film depends on contents of diamond ( $\text{sp}^3$ ) distributed in the film. There are mainly two content —  $\text{sp}^3$  and  $\text{sp}^2$  (graphite) existed in the diamond film. The more  $\text{sp}^3$  compared with  $\text{sp}^2$  in the film, the better of optical properties (transmission and refractive index) will

be. Commonly used method to judge relative proportion of

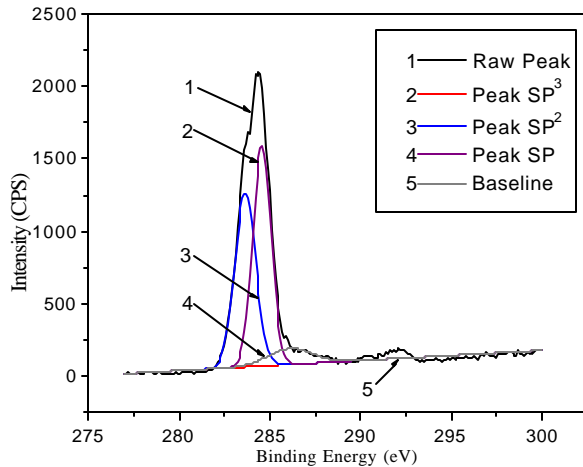


Fig.1 Measurement result of X-ray photoelectric spectroscopy before FIB processing

$sp^3$  and  $sp^2$  in diamond film is X-ray photoelectric spectroscopy (XPS). Fig.1 and Fig.2 show the XPS spectrum over the coated windows before and after FIBM, in which curves 2,3 and 4 are deconvolution's of curve 1 (raw peak). We observe a sharp diamond ( $sp^3$  bonded carbon) peak represented by curve 2 is higher than curve 3 ( $sp^2$ ) before FIB processing, and it is much higher after FIB processing, indicating a very high diamond-phase content. The possible reason is that some compound of graphite is transferred to diamond during bombing of  $Ga^+$  with high energy during FIBM. In other words, FIBM is helpful for diamond film processing and machining. The influence of the FIBM on content of  $sp^3$  in the film during the milling process can be ignored.

#### B. Optical properties after FIBM

More or less of  $Ga^+$  implantation may occur during FIBM that will affect transmission of the film due to remained  $Ga^+$  implanted on the surface of the film, especially for visible wavelength. Fig.3 is transmission comparison of the diamond

film measured before and after FIB processing measured by

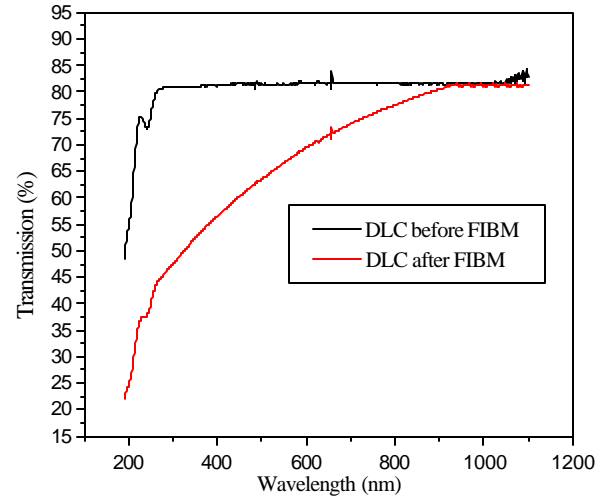


Fig.3 Transmission comparison of diamond film measured before and after FIB processing with ion

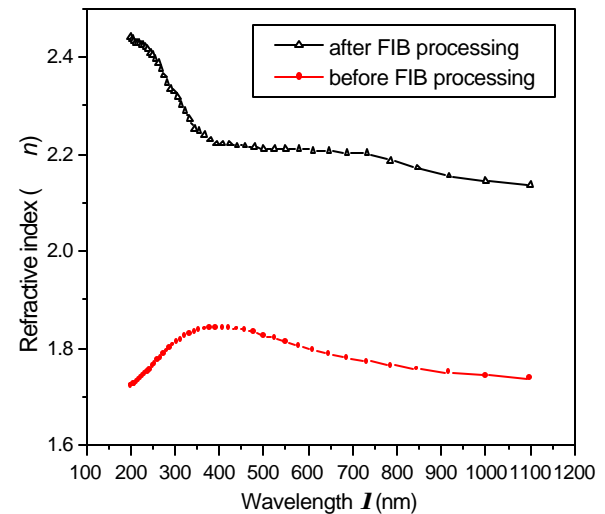


Fig.4 Refractive index comparison of DLC film before and after FIB processing

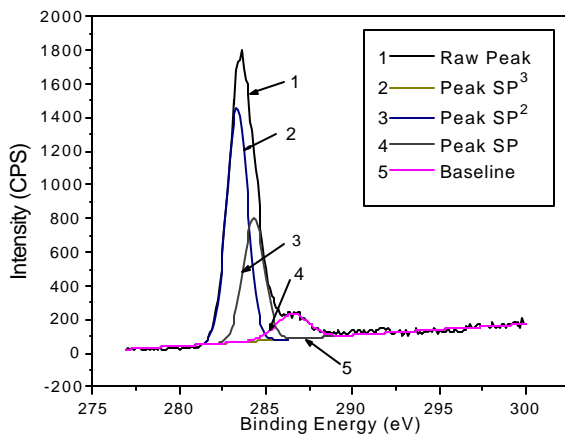


Fig.2 XPS measurement results after FIB processing

use of UV-VIS transmittance spectrum (UV-VIS spectrophotometer HP8453). It can be seen that variation of transmission before and after FIBM is a few in the region of near infrared wavelength. Transmission is stable with value of about 81% for wavelength larger than 900 nm that can meet requirement of conventional optical system. The film after FIBM is more suitable for infrared applications.

Fig.4 is refractive index versus wavelength before and after FIBM process measured by use of ellipsometer (Mizojiri Kogaku) equipped with He-Ne laser. It can be seen that the refractive index increases after the FIBM. The increment after FIBM is caused due to more or less  $Ga^+$  implantation under ion energy of 50 keV. Fig.5 is simulated results of implanted depth of  $Ga^+$  versus different ion energy by use of commonly used ion beam simulation software (SRIM 2000). It can be seen that the implantation depth under the diamond-like surface is 313 Angstrom corresponding to ion energy of 50 keV. The increased refractive index is still usable for

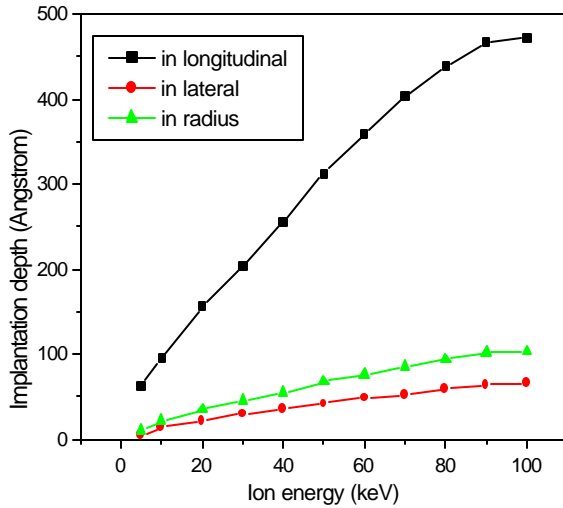


Fig.5 Simulation results of ion implantation depth vs. ion energy in directions of longitudinal, lateral and radius.

conventional optical system. However, the lens should be

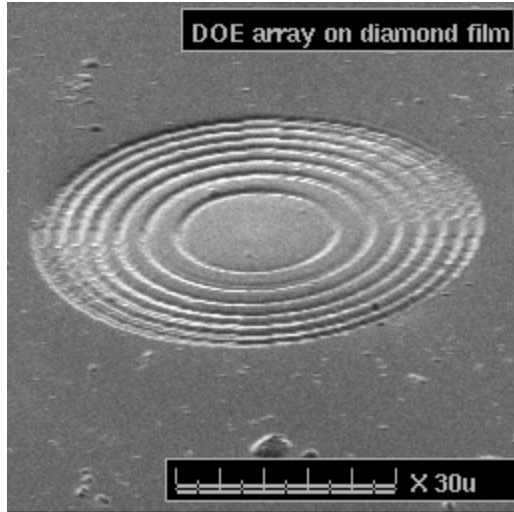


Fig.6. SEM micrograph of the DOE milled by use of FIB on diamond film with thickness of 1.5  $\mu\text{m}$ .

design in terms of the new refractive index of the corresponding designed wavelength.

Therefore, we can draw conclusion that the film characteristic is stable during FIBM process because it is low temperature machining process. The side-effect —  $\text{Ga}^+$  implantation with ion energy of 50 keV is not large enough to apparently affect the transmission of the film in infrared region. Influence of FIBM on optical characteristics of the diamond film can be ignored.

#### IV. APPLICATION EXAMPLE

As an application example, we fabricate a diffractive optical element with six annulus on the diamond-like film. The relief accuracy is affected by beam spot size and pixel overlapping during raster scan. The relief accuracy will be deteriorated by

use of larger beam spot size and smaller pixel overlapping. Considering the default discrete step size of 0.5  $\mu\text{m}$ , we use the 100nm beam spot size and 60% overlapping. Comparing the effects of height error and lateral error on the relief accuracy, the lateral error is prominent. Because of line broadening effect caused by wing of Gaussian distribution of the ion beam, the actual milled linewidth is larger than designed size (called line broadening). Considering this, the defined linewidth should be less than the designed size. Concrete value depends on chosen of beam spot size, beam current density and calibration of ion dose. It takes ~20 minutes to fabricate a single micro-DOE using the FIBM.

The relief accuracy is determined by beam spot size and overlapping of pixels during the raster scan. The larger the beam spot size and the smaller pixels overlapping, the worse the relief accuracy will be. Considering the used interline step of 0.5  $\mu\text{m}$ , 100 nm beam spot size and 60% overlap were used for our processes.

Fig.6 is micrograph of scanning electron microscopy (SEM) for the fabricated DOE. We utilized an AFM (Nanoscope model III A from Digital Instruments) to analyze the surface roughness before and after FIB milling. The scanner was calibrated with a 160 nm height standard. Probing a  $1 \times 1 \mu\text{m}^2$  region, an average roughness,  $R_a$ , are 2.22 nm and 2.37 nm for the 1.5  $\mu\text{m}$  thick diamond film before and after FIBM, respectively.

In order to evaluate optical performance of the fabricated element, diffraction efficiency of the DOE is measured by use of tunable laser (operating wavelength of 1550nm), objective lens (10 $\times$ ), collimation lens, and photodetector (InGaAs), is 73%, which is acceptable for conventional use. It reflects from other side that the influence of FIBM for the film is a little and can be neglected.

#### V. SUMMARY

In summary, fabrication of DOE on diamond film by use of FIBM is valid. Surface roughness after milling is suitable for normal optical application. No compound of diamond changes to graphite after FIBM. Influence of FIBM upon transmission and refractive index of diamond film is a little, and can be neglected in practical application.

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**Fu Yongqi:** was born in May 1967. He received his B.Eng, M.S(Eng), Ph.D in mechanical engineering from Jilin University of Technology, Changchun Institute of Optics and Fine Mechanics, Chinese Academy of Sciences in 1988, 1994, and 1996 respectively. He worked in State Key Laboratory of Applied Optics from 1996 to 1998 as a Postdoctoral Fellow. And then, worked in Precision Engineering and Nanotechnology (PEN) Center as Research Fellow from Aug. 1998 to Dec. 2001. He is leader of FIB group in PEN Center. Currently, he works in Singapore-MIT-Alliance, IMST program as Research Fellow. His Research direction is application of FIB in microelectronics, microfabrication, nano-optoelectronics, MOEMS, micro-optics, fiber optics, and optical measurement. He is the first author of more than 50 referred journal papers.

**Bryan Kok Ann Ngoi:** heads of Manufacturing Engineering, director of PESRP, School of Mechanical and Production Engineering, Nanyang Technological University. He received his B.Eng degree (with honors) in 1985 from National University of Singapore and his Ph.D degree in 1990 from the University of Canterbury. His research interests include precision measurements, design for manufacturing, metrology and inspection, geometric dimensioning and tolerancing, fixture design, and micromachining. Dr. Ngoi is the author of more than 120 papers, including 80 international referred journal papers.